



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 1 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### CONTENTS

1.0	SCOPE AND APPLICATION
2.0	METHOD SUMMARY
3.0	SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
4.0	INTERFERENCES AND POTENTIAL PROBLEMS
5.0	EQUIPMENT/APPARATUS
6.0	REAGENTS*
7.0	PROCEDURE
7.1	Glassware Cleaning Procedures
7.2	Water Sample Digestion
7.3	Solid Sample Digestion
7.4	Total Solids
7.5	ICP Operation*
8.0	CALCULATIONS
8.1	Sample Concentration
8.2	Laboratory Control Sample
8.3	Matrix Spike/Matrix Spike Duplicate
9.0	QUALITY ASSURANCE/QUALITY CONTROL
9.1	Initial Calibration Verification
9.2	Interference Check Standards
9.3	Continuing Calibration Verification
9.4	Initial/Continuing Calibration Blanks
9.5	Method Blank
9.6	Laboratory Control Sample
9.7	Matrix Spike/Matrix Spike Duplicate
9.8	Linear Analytical Range
9.9	Serial Dilution
9.10	Dilution Analysis
9.11	Initial Demonstration of Capability



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 2 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

---

#### CONTENTS (Cont'd)

- 9.12 Method Detection Limit Studies
- 9.13 Reporting Limit Standards\*
- 9.14 Instrument Detection Limits
- 9.15 System Troubleshooting
- 9.16 Nonconformance Memo
  
- 10.0 DATA VALIDATION
  
- 11.0 HEALTH AND SAFETY
  
- 12.0 REFERENCES
  
- 13.0 APPENDICES
  - A - Tables\*
  - B - TJA ICAP-61E Daily Operation Procedure
  - C - Thermo Elemental ICAP-61E Trace Analyzer Daily Operation Procedures
  - D - Attachments\*

\* These sections affected by Revision 3.0

SUPERCEDES: SOP #1811; Revision 2.0; 01/23/06; U.S. EPA Contract EP-C-04-032

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 3 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

---

#### 1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analysis procedures for metals in water and solid matrices using inductively coupled plasma (ICP) atomic emission spectroscopy. This method is based on Environmental Protection Agency (EPA) Methods SW846/3015/3050B/ 6010B and those requirements set forth in the latest approved version of the National Environmental Laboratory Accreditation Committee (NELAC) Quality Systems section. A list of target analyte list (TAL) compounds routinely analyzed by the Response Engineering Analytical Contract (REAC) Laboratory and the corresponding reporting limits (RLs) are provided in Table 1, Appendix A. These RLs are dependent upon the ICP instrumentation and operating parameters used (i.e., plasma power, sample aspiration method, and support gas flow rate), sample preparation method, and the sample matrix. Typical calibration standard concentrations are listed in Table 2, Appendix A. To ensure valid data, the analyst should be familiar with ICP spectroscopic techniques for correction of spectral, chemical, and physical interferences which can adversely affect the analysis.

This method may not be changed without the expressed approval of the Inorganic Group Leader, the Analytical Section Leader and the Quality Assurance Officer (QAO). Only those versions issued through the REAC document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

#### 2.0 METHOD SUMMARY

A representative 45-milliliter (mL) aqueous sample is digested in 5 mL of concentrated nitric acid ( $\text{HNO}_3$ ) in a Teflon® digestion vessel using microwave heating. After the digestion process, the sample is cooled, and then filtered or allowed to settle in a clean sample bottle prior to analysis.

A representative 1 to 2 gram (wet weight) sample is digested with repeated additions of nitric acid ( $\text{HNO}_3$ ) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Hydrochloric acid ( $\text{HCl}$ ) is added to the digestate and the sample is refluxed. The digestate is then diluted to a final volume of 100 milliliters (mL) with deionized (DI) water.

Simultaneous or sequential, multi-element determination of metals in solution may be achieved using ICP spectroscopic techniques. The method measures element-specific light emitted by the metals in the sample. Sample solutions are nebulized and the resultant aerosol is transported to the plasma torch where the spectrum of analyte-specific atomic-line emissions is produced. The spectrum is dispersed by a diffraction grating and the intensities of the individual emission lines are monitored by photomultiplier tubes. Background correction is required for trace element determination and is measured at wavelengths adjacent to the analyte lines during analysis. The positions selected for background intensity measurement are determined by the complexity of the spectrum adjacent to the analyte lines. These positions must be free of spectral interference and must reflect the same change in background intensity as that occurring at the analyte lines. The possibility of additional matrix-dependent interelement effects should be recognized and appropriate corrections made.

#### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Sample holding times, suggested collection amount, preservative, and type of containers are as follows:

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 4 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

Measurement Parameter	Volume/Weight Required	Type of Containers	Preservative	Holding Time
<u>Metals</u> (except hexavalent chromium and mercury):				
Total Recoverable	1000 mL	P, G	HNO <sub>3</sub> to pH < 2	6 months
Dissolved	1000 mL	P, G	Filter on site HNO <sub>3</sub> to pH < 2	6 months
Suspended	1000 mL	P, G	Filter on site	6 months
Total	1000 mL	P, G	HNO <sub>3</sub> to pH < 2	6 months
Solids	200 g	G	none/4 °C	6 months

P - plastic, G - glass, g - grams, mL - milliliters

#### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

During the microwave process, water samples that contain organics will result in higher vessel pressures that have the potential to cause venting of the digestion vessels. This can result in loss of analytes and/or sample. A smaller volume of sample diluted to 45 mL may be used but the dilution must be taken into account during the final calculation of analyte concentration.

Solid samples may contain diverse matrix types. Spiked samples and other relevant quality control (QC) samples may aid in determining if there are any interferences.

Spectral interferences are the most common type of interference in ICP spectroscopy. They are caused by: 1) interelement effects due to overlap of an emission line from another element or stray light from the line emission of high concentration elements, and 2) background contribution from continuum or recombination events. Interelement effect corrections (IECs) compensate for overlap or stray light spectral interferences. Background contributions are compensated by background correction measurements adjacent to the analyte lines.

Physical interferences are associated with the sample nebulization or transport process. Changes in viscosity and surface tension may cause significant inaccuracies, especially for samples containing high dissolved solids or high acid concentrations. When physical interferences are present, they can be reduced by diluting the sample or by using a peristaltic pump.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant for ICP spectroscopic techniques. If observed, they can be minimized by careful selection of instrument operating parameters, buffering the sample, and matrix matching. Chemical interferences are highly dependent upon the specific element and sample matrix type.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 5 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### 5.0 EQUIPMENT/APPARATUS

- Inductively Coupled Plasma Spectrometer, computer-controlled sequential or simultaneous emission spectrometer with interelement and background correction capabilities, and provisions for interfacing to a printer and an autosampler
- Microwave Digestion System, with temperature control and rotating turntable, well ventilated with corrosion-resistant cavity
- Hot block digestion system or hot plate, temperature-controlled
- Electronic Variable Volume Autopipettors, BIOHIT 500 to 5000 microliters ( $\mu$ L) and BIOHIT 100 to 1000  $\mu$ L or equivalent
- Volumetric flasks, Class A, varying volumes
- Microwave digestion vessels for water samples, Teflon, capable of holding ~75 milliliters (mL), designed for temperatures up to 260°C with self-regulating pressure control
- Digestion vessels for soil samples, capable of holding ~250 mL
- Watch glasses or vapor recovery device
- Glass dispensers, 2-liter (L), 1-L, or 1-gallon, checked quarterly for accuracy
- Graduated Cylinder, Class A, 50 mL
- Volumetric flasks, Class A, assorted volumes
- Balance, top-loading, capable of reading to 0.01 grams (g), for weighing digestion vessels before and after digestion
- Henke SASS plastic syringes or equivalent
- Corning SCFA 0.45 microns ( $\mu$ m) filters or equivalent

#### 6.0 REAGENTS

- Deionized (DI) water, Type I Water (American Society for Testing and Materials [ASTM] D1193), for the preparation of all reagents and calibration standards and as dilution water
- Nitric acid, concentrated, trace metal grade
- Nitric Acid, 10 percent (%) volume to volume (v/v), for the preparation of working standards, also to be used for the Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 6 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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- Hydrochloric Acid, concentrated, trace metal grade
- Hydrogen Peroxide, 30 percent (%), reagent or trace grade
- Argon Plasma Support Gas, commercially available, in pressurized cylinders or from the gas outlet of a liquid argon dewar
- Stock Calibration Standards, 1000 milligrams per liter (mg/L), commercially available, accompanied by a certificate of analysis, for all elements except aluminum (Al), barium (Ba), calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), sodium (Na), and vanadium (V)
- Stock Calibration Standards, 10,000 mg/L, commercially available, accompanied by a certificate of analysis, for Al, Ba, Ca, Fe, Mg, K, Na, and V

NOTE: Stock standard solutions may also be prepared from high purity metals, oxides, or non-hygroscopic reagent-grade salts using Type I water and trace metal grade HNO<sub>3</sub>. Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements.

- Intermediate Calibration Standard, 100 mg/L - Dilute 10 mL of the 1000 mg/L stock calibration standards for beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), manganese (Mn), nickel (Ni), silver (Ag) and zinc (Zn) to 100 mL in a Class A volumetric flask using 10% nitric acid
- Intermediate Calibration Standard, 1000 mg/L - Dilute 10 mL of the 10000 mg/L stock calibration standards for Ba and V to 100 mL in a Class A volumetric flask using 10% nitric acid
- Working Calibration Standard - Prepare 200 mL of this working calibration standard in 10% nitric acid using the volumes listed in Table 2.
- Water Reporting Limit (RL) Standard (RLW) - Dilute 0.5 mL of the Water RL Spiking Solution to 500 mL in a Class A volumetric flask using 10% nitric acid.
- Soil RL Standard (RLS) - Dilute 0.5 mL of the soil RL Spiking Solution to 500 mL in a Class A volumetric flask using 10% nitric acid.

NOTE: Standard Preparation Logs for preparing the water or soil RL Spiking Solutions are in Attachment 1, Appendix D.

- Stock Initial Calibration Verification/Continuing Calibration Verification Standard (ICV/CCV), 100 mg/L containing all analytes, commercially available, accompanied by a certificate of analysis
- Stock ICV/CCV, 5000 mg/L of Al, Ca, Fe, Mg, K, and Na, commercially available, accompanied by a certificate of analysis

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 7 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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- Working ICV/CCV containing all analytes at 2 mg/L except Al, Ca, Fe, Mg, K, Na at 20 mg/L - Add 10 mL of the stock 100 mg/L ICV/CCV and 1.8 mL of the stock 5000 mg/L ICV/CCV to 500 mL using 10% nitric acid in a Class A volumetric flask.
- Working ENDCCV containing all analytes at 2.5 mg/L except Al, Ca, Fe, Mg, K, Na at 12.5 mg/L - Add 12.5 mL of the stock 100 mg/L ICV/CCV and 1 mL of the stock 5000 mg/L ICV/CCV to 500 mL using 10% nitric acid in a Class A volumetric flask.
- MS/MSD Spiking Solution - Prepare spiking solutions in 10% nitric acid at the concentrations listed in the Spiking Solution Log in Attachment 2, Appendix D.
- Laboratory Control Sample (LCS), commercially available, accompanied by a certificate of analysis, or a blank matrix spiked with the target analytes from an independent source at or near mid-range of the calibration. Prepare the LCS with each analytical batch of samples.
- Stock Interference Check Standard (ICS) containing interferents (INT-A1) - Al, Ca, Mg at 5000 mg/L and Fe at 2000 mg/L, commercially available, accompanied by a certificate of analysis
- Stock ICS containing analytes (INT-B3) - Cd, Ni, Zn at 100 mg/L; Sb at 60 mg/L; Ba, Be, Cr, Co, Cu, Mn, V at 50 mg/L; Ag at 20 mg/L; As, Tl, at 10 mg/L; Pb, Se at 5 mg/L, commercially available, accompanied by a certificate of analysis
- Working ICSA - Add 20 mL of the stock INT-A1 standard to 200 mL using 10% nitric acid in a Class A volumetric flask
- Working ICSAB - Add 20 mL of the stock INT-A1 standard and 2 mL of the stock INT-B3 standard to 200 mL using 10% nitric acid in a Class A volumetric flask
- Internal Standard solution - Add 5 mL of stock 1000 mg/L yttrium (Y) and 9.93 g of stock Lithium Nitrate ( $\text{LiNO}_3$ ) to 1000 mL using 10% nitric acid in a 1-liter polyethylene bottle. NOTE: 1000 mg/L Y and reagent grade  $\text{LiNO}_3$  are commercially available.

**NOTE:** Premixed certified standards will be stored according to the manufacturer's documented storage requirements. These standards may be kept in storage up to the manufacturer's stated expiration date. Once dilutions are made, the standards will be stored for a period not to exceed six months or the manufacturer's expiration date, whichever is less.

**NOTE:** All calibration standards and spiking solutions will be prepared and documented in accordance with REAC SOP #1012, *Preparation of Standard Solutions and Reagents*.

**NOTE:** Stock concentrations may vary depending on the vendor; thus, the concentration of intermediate standards may also vary. Preparation instructions may differ based on the volumes prepared and the concentrations of standards available. The final working standard concentrations will remain as stated in the above section.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 8 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### 7.0 PROCEDURE

##### 7.1 Glassware Cleaning Procedures

All glassware and Teflon containers should initially be cleaned using the following sequence: detergent, tap water, 1:1 nitric acid, tap water, and Type I water. If it can be documented through an active analytical quality control (QC) program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

##### 7.2 Water Sample Digestion

1. Weigh the empty digestion vessel, valve and cap to 0.01 g . Record the weight on the sample digestion log.
2. Check the pH of the aqueous sample to ensure that the pH is <2. Record the pH on the sample digestion log. If the pH is not <2, adjust the sample pH to <2 with additional concentrated HNO<sub>3</sub>. Do not add more than 1% of the total volume of sample. Note the volume of HNO<sub>3</sub> added on the sample digestion log. A nonconformance memo must be written if pH is greater than or equal to 2.0 prior to adjustment.
3. Measure 45 mL of a well mixed sample in a Class A graduated cylinder. Transfer the sample to a digestion vessel and mark the digestion vessel with the sample number.
4. Measure 45 mL of DI water in a Class A graduated cylinder and transfer to a digestion vessel. Mark the digestion vessel as the method blank. One method blank must be prepared for each analytical batch not to exceed 20 samples.
5. Measure 45 mL of the LCS in a Class A graduated cylinder and transfer to a digestion vessel. Mark the digestion vessel as the LCS. One LCS must be prepared for each analytical batch not to exceed 20 samples.
6. Measure two separate well-mixed 45-mL portions of the sample chosen for the MS/MSD in a Class A graduated cylinder. Transfer these portions of samples to separate digestion vessels, add 0.5 mL of the MS/MSD spiking solution to each and mark the digestion vessels as the MS and MSD. At least one MS/MSD must be digested with every 10 samples or per project.
7. Add 5 mL of concentrated HNO<sub>3</sub> to each vessel. Check to make sure that the pressure relief disks are in the caps and hand tighten the caps on the vessels. Weigh each digestion vessel and record the weight on the sample digestion log.
8. Evenly distribute the vessels in the carousel according to the manufacturer's instructions. When fewer than the recommended number of vessels are digested, be sure to include additional vessels with the same volume of DI water and concentrated HNO<sub>3</sub> as the samples.



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 9 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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9. Program the microwave digestion unit according to the manufacturer's recommended specifications to bring the samples to  $160 \pm 4$  degrees Centigrade ( $^{\circ}\text{C}$ ) in 10 minutes and rise slowly to between 165 and  $170^{\circ}\text{C}$  in the second 10 minutes. Power settings may be adjusted as long as they result in the same time and temperature conditions.
10. At the end of the digestion, allow the digestion vessels to cool for a short period of time in the microwave and then on the bench or in a water bath. Once the vessels are cool, weigh and record the weight of each digestion vessel assembly. If the weight of the digestate has decreased more than 10%, discard the sample.
11. Uncap and vent each vessel in a fume hood and transfer the sample to an acid-cleaned sample bottle. If the digested sample contains particulates, filter prior to analysis using the plastic syringes and the  $0.45\ \mu\text{m}$  filters, or equivalent.
12. Correct the concentration values from analysis by a dilution factor of 1.11 (45 mL sample + 5 mL of acid/45 mL of original sample)

#### 7.3 Solid Sample Digestion

1. Mix the sample thoroughly. Weigh to the nearest 0.01 grams (g) and transfer a 1 to 2 g sample to a digestion vessel and mark the digestion vessel with the sample number.
2. Mark a separate digestion vessel as the method blank. One method blank must be prepared for each analytical batch not to exceed 20 samples.
3. Weigh 1.0 g of the LCS and transfer to a digestion vessel. Mark the digestion vessel as the LCS. One LCS must be prepared for each analytical batch not to exceed 20 samples.

NOTE: A blank spike (e.g. sand spiked with target analytes from an independent source at or near mid-range of the calibration) may be used in lieu of the LCS.

4. Weigh two separate well-mixed portions of the sample chosen for the MS/MSD and transfer these portions to separate digestion vessels. Add 2.0 mL of the MS/MSD spiking solution to each and mark the digestion vessels as the MS and MSD. At least one MS/MSD must be digested with every 10 samples or per project.
5. Add 10 mL of 1:1  $\text{HNO}_3$  to each digestion vessel and cover with a watch glass or vapor recovery device. Heat the sample to  $95 \pm 5$  degrees Centigrade ( $^{\circ}\text{C}$ ) and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated  $\text{HNO}_3$ , replace the cover and reflux for 30 minutes. If brown fumes are generated during heating, add concentrated  $\text{HNO}_3$  in 5-mL increments until no brown fumes are observed. Cover the sample with a watch glass or vapor recovery device and continue heating the digestate at  $95 \pm 5^{\circ}\text{C}$  without boiling for a total of two hours. Maintain solution over the bottom of the

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 10 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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vessel at all times.

6. Cool the sample. Add 2 mL of DI water, 3 mL of 30%  $H_2O_2$ , cover the digestion vessel with a watch glass and heat until the effervescence subsides. Cool the sample.
7. Continue to add 30%  $H_2O_2$  in 1-mL increments with warming until the effervescence is minimal or the sample appearance remains unchanged. Do not add more than a total of 10 mL of 30%  $H_2O_2$ .
8. Cover the sample with a watch glass and continue heating the digestate until the volume has been reduced to approximately 5 mL or heat at  $95 \pm 5^\circ C$  without boiling for two hours.
9. Cool the sample. Add 10 mL of concentrated HCl to sample digestate and cover with a watch glass. Place on heating source and reflux at  $95 \pm 5^\circ C$  for 15 minutes.
10. After cooling, dilute to 100 mL with DI water. Remove particulates by filtering prior to analysis.
11. Correct the concentration values from analysis using the sample weight, final volume and % total solids.

#### 7.4 Total Solids

Total solids analysis will be conducted in accordance with REAC SOP #1843, *Determination of Total Solids in Solid Samples*.

#### 7.5 ICP Operation

Differences between the various makes and models of satisfactory ICP spectrometers preclude the formation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for a particular instrument. Daily operation and troubleshooting procedures for the Thermal Jarrell Ash (TJA) ICAP-61E and Thermo Elemental ICAP-61E Trace Analyzer spectrometers are in Appendix B and Appendix C, respectively.

In general, after igniting the plasma, the instrument should be allowed to thermally stabilize for 30 to 60 minutes prior to calibration. After the plasma has stabilized, the instrument profile for copper (Cu) or another suitable analyte line [eg, arsenic (As) for 61E Trace] must be verified and the spectrometer must be calibrated according to the manufacturer's recommended procedures.

1. Calibrate the ICP using a blank and the working calibration standard (high standard)
2. Immediately following calibration, analyze the ICV standard. The percent recovery must be within  $\pm 10\%$ .
3. Immediately following the ICV, analyze the ICB. The concentration must be less than ( $<$ )

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 11 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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the reporting limit (RL) for each element.

4. Analyze the appropriate RL standard (RLW or RLS), ICSA, ICSAB, continuing calibration verification (CCV), and continuing calibration blank (CCB) standards. Refer to Section 9.0 for QC criteria.
5. Analyze the method blank, LCS, and samples.
6. A CCV/CCB must be run every 10 samples. At the end of the analytical sequence, a ENDCCV/CCB must be analyzed.
7. The ICSA and ICSAB must be run at the beginning of the sequence and when the sequence exceeds an 8-hour shift.
8. If the concentration found is greater than the linear analytical range (Section 9.8), the sample must be diluted with 10% HNO<sub>3</sub> and reanalyzed (Section 9.10).
9. To verify the absence of interference, follow the serial dilution analysis procedure in Section 9.9.

#### 8.0 CALCULATIONS

##### 8.1 Sample Concentration

For the determination of metal concentration in solution, the value in milligrams per liter (mg/L) for the ICAP61E or micrograms per liter (µg/L) for the ICAP Trace is read directly from the read-out system of the instrument. For aqueous samples, report concentrations as µg/L, where, µg/L = mg/L x 1000.

If a dilution of the sample is required:

$$\text{concentration of metal in sample} = A \times \frac{C+B}{C}$$

where:

A	=	mg/L (or µg/L) of metal in diluted aliquot from read-out
B	=	Acid blank matrix used for dilution, mL
C	=	Sample aliquot, mL

For solid samples, all concentrations are calculated as mg/kg based on wet weight; therefore:

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 12 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

$$mg\ metal/kg\ sample = \frac{A \times V}{F \times W} \times DF$$

where:

A = mg/L (or µg/L) of metal in processed sample from read-out  
F = concentration unit factor (1.00 for ICAP61E, 1000 for ICAP Trace)  
V = Final volume of the processed sample, mL  
W = Weight of sample, grams  
DF = Dilution factor for diluted samples (1.00 with no sample dilution)

If dilution of the sample is required, DF is given by:

$$DF = \frac{C+B}{C}$$

where:

B = mL of acid blank matrix used for dilution  
C = mL of sample aliquot

For solid samples, report concentrations as mg/kg based on dry weight:

$$mg\ metal/kg\ sample\ (dry\ basis) = \frac{mg/kg\ (wet\ basis)}{(S / 100)}$$

where:

S = percent total solids in the sample

#### 8.2 Laboratory Control Sample

Percent recovery (%R) must be within 75-125% and calculated as:

$$\%R = \frac{(LCS - B)}{SA} \times 100$$

where:

LCS = LCS result, µg/L or mg/kg  
B = Method blank result, µg/L or mg/kg  
SA = Spike added, µg/L or mg/kg

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 13 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### 8.3 Matrix Spike/Matrix Spike Duplicate Sample

Spike sample percent recovery (%R) must be within 75-125% and calculated as:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

where:

SSR = Spiked sample result  
SR = Sample result  
SA = Spike added

The Relative Percent Difference (RPD) of matrix spike and matrix spike duplicate samples should be within  $\pm 20\%$  and calculated as:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where:

S = %R for matrix spike sample  
D = %R for matrix spike duplicate sample

#### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

All QC data should be maintained and available for easy reference and inspection.

##### 9.1 Initial Calibration Verification

The calibration curve must be verified by the analysis of an ICV standard (at or near mid-range) from an independent source. The ICV result must be within 10% of the true value for the calibration to be considered valid. If the ICV is outside QC limits, the instrument must be re-calibrated.

##### 9.2 Interference Check Standards

ICP interelement corrections are verified by analyzing ICSs at the beginning and each time the analytical run exceeds an 8-hour shift. Results must be within  $\pm 20\%$  of the true value for each element.

##### 9.3 Continuing Calibration Verification

The working standard curve must be verified by analyzing the CCV standard (at or near the mid-range) after every 10 samples and the ENDCCV at the end of the sequence. CCV/ENDCCV results must be within  $\pm 10\%$  of the true value, or the previous ten samples must be reanalyzed.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 14 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### 9.4 Initial/Continuing Calibration Blanks

The ICB/CCB results must be less than the reporting limit (RL) for the sequence to continue.

#### 9.5 Method Blank

A method blank must be prepared for each analytical batch of samples (not to exceed 20 samples) of the same matrix. The method blank results must be less than the RL. A method blank containing an analyte concentration >RL may be used in instances when the sample concentrations are at least 10 times the method blank concentration.

#### 9.6 Laboratory Control Sample

Aqueous and solid LCSs must be obtained from an independent source, and must be prepared with each analytical batch of samples using the same preparation method as that employed for the samples with the frequency of 1 in 20 samples per matrix. The LCS sample may be either a certified reference material or a blank matrix spiked with the target analytes from an independent source at or near mid-range of the calibration. LCS results for each analyte must be within the specifications supplied by the vendor or within 75 - 125% of the true value and are calculated as in Section 8.2.

#### 9.7 Matrix Spike/Matrix Spike Duplicate

At least one MS and one MSD sample must be digested with every 10 samples of the same matrix, or with each project type to verify the accuracy of the method. In the event there is not sufficient sample available in the batch to run a MS/MSD, a LCS/laboratory control sample duplicate (LCSD) must be run. Recoveries are calculated as in Section 8.3.

The Relative Percent Difference (RPD) of MS/MSD samples must be within  $\pm 20\%$  and calculated as in Section 8.3.

#### 9.8 Linear Analytical Range

One or more linear analytical range (LAR) standards must be analyzed to determine the maximum linear range of the calibration for each element. Recovery must be within 90-110 %. If the recovery is outside these limits, the maximum calibration standard concentration defines the linear range. LAR standards must be analyzed and reported on a quarterly basis.

#### 9.9 Serial Dilution

A sample (typically the sample chosen for the MS/MSD) from each project in an analytical batch is analyzed at a 5x dilution in conjunction with the samples. The concentration in the undiluted sample must be greater than or equal to 50x the IDL to obtain a meaningful comparison. The results of the serial dilution are multiplied by the dilution factor and compared to the original determination (undiluted sample). Agreement within  $\pm 10\%$  between the concentrations for the undiluted sample and the diluted sample indicates the absence of matrix interferences for undiluted samples meeting the 50x

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 15 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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IDL criteria.

If the concentration of all analytes in all samples is less than 50x the IDL, serial dilution is not performed. Samples may also be successively diluted and analyzed to eliminate interferences. These samples will be identified as dilution samples and not as serial dilutions.

#### 9.10 Dilution Analysis

If the concentration of any analyte in any sample exceeds the linear range, the sample must be diluted and re-analyzed. An appropriate dilution or series of dilutions (for example, 5x, 10x, 20x) may be required depending on the concentration in the undiluted sample. Results are reported from the lowest dilution that falls within the linear range.

If chemical/physical matrix effects are suspected or for analytes that saturate the detector, samples must be diluted and re-analyzed. An appropriate dilution or series of dilutions may be required depending on the concentrations in the undiluted sample. Comparisons are first made with respect to the undiluted sample and then, within the series. Based on the analyst's professional judgement, results are reported from the diluted sample that has the smallest dilution factor and indicates the absence of interferences.

An optional approach to determine if chemical/physical matrix effects are present is to use post digestion spike (PDS) analysis.

#### 9.11 Initial Demonstration of Capability

Initial proficiency in ICP analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicates of a well-mixed reference standard is analyzed using the procedures outlined in this SOP. Calculate the average mean and standard deviation (s) in  $\mu\text{g/L}$  for waters and  $\text{mg/kg}$  for soils. The QAO will tabulate the results from all of the analysts per matrix per parameter, and calculate control limits.

#### 9.12 Method Detection Limit Studies

Method detection limit (MDL) studies will be run on an annual basis for each ICP instrument for the water and soil matrix to verify the minimum concentration that can be measured and reported with 99% confidence. A minimum of seven replicates will be used for the study (EPA 1984).

#### 9.13 Reporting Limit Standards

The RLW standard is run each time water samples are analyzed by the ICP. The RLS standard is run each time soil samples are run on the ICP. The RLS and RLW upper and lower control limits are established based on the soil and water MDL study results.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 16 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### 9.14 Instrument Detection Limits

IDLs are run on a quarterly basis on each ICP and are used as the basis for the serial dilution analysis.

#### 9.15 System Troubleshooting

Refer to Appendix B and C for instrument operation and troubleshooting.

#### 9.16 Nonconformance Memo

A nonconformance memo will be generated any time an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the Quality Assurance Officer for verification of corrective action.

### 10.0 DATA VALIDATION

Data will be assessed based on the project's data quality objectives by the Data Validation and Report Writing Group using the most current revision of the REAC SOP #1017, *Data Validation Procedure for Routine Inorganic Analysis*. However, data is considered satisfactory for submission purposes when all of the requirements listed in this procedure are met.

### 11.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for following the chemical hygiene plan and laboratory safety program regarding the safe handling of the chemicals specified in this method.

When working with potentially hazardous materials, refer to EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to REAC SOP #3013, *REAC Laboratory Safety Program*.

### 12.0 REFERENCES

Thermo Jarrell Ash Corporation. 1991. *ICAP-61E Spectrometer Operator's Manual*.

Thermo Elemental. 2001. *ICAP-61E Trace Analyzer Operator's Manual*.

National Environmental Laboratory Accreditation Committee (NELAC), *Quality Systems*, current approved version.

U.S. Environmental Protection Agency. 1984. Federal Register, 40 Code of Federal Regulations (CFR) Part 136, Appendix B, *Definition and Procedure of the Determination of the Method Detection Limit - Revision 1.11*, October 26, 1984.





## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 17 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 1994. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Method 3015.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 1996. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Method 3050B.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 1996. *Test Methods for Evaluating Solid Waste*, SW-846, 3rd ed., Method 6010B.

#### 13.0 APPENDICES

- A. Tables
- B. TJA ICAP-61E Daily Operation Procedures
- C. Thermo Elemental ICAP-61E Trace Analyzer Daily Operation Procedures
- D. Attachments



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 18 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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APPENDIX A  
Tables  
SOP #1811  
March 2006

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 19 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

TABLE 1. ICP Detection and Reporting Limits for TAL Compounds							
ANALYTE	TYPICAL ANALYTICAL RANGE (mg/L)	TYPICAL INSTRUMENT DETECTION LIMIT (IDL, mg/L)		TYPICAL REPORTING LIMITS			
				ICAP61E <sup>(1)</sup>		Trace <sup>(2)</sup>	
		ICAP 61E	Trace	Soil (mg/kg)	Water (mg/L)	Soil (mg/kg)	Water (mg/L)
Aluminum	0.040 - 500	0.02	0.010	18	0.05	25	0.040
Antimony	0.014 - 100	0.03	0.004	6	0.05	1.4	0.014
Arsenic	0.015 - 100	0.04	0.004	8	0.08	2	0.015
Barium	0.002 - 25	0.0005	0.001	1	0.005	0.3	0.002
Beryllium	0.002 - 25	0.0005	0.001	0.5	0.002	0.3	0.002
Cadmium	0.002 - 50	0.002	0.001	0.5	0.005	0.4	0.002
Calcium	0.063 - 1000	0.010	0.01	50	0.10	20	0.063
Chromium	0.002 - 100	0.003	0.001	0.5	0.005	0.4	0.002
Cobalt	0.003 - 100	0.003	0.001	1	0.01	0.3	0.003
Copper	0.004 - 100	0.002	0.001	1	0.01	0.4	0.004
Iron	0.025 - 100	0.005	NA	10	0.025	NA	NA
Iron 2599	0.025 - 100	NA	0.005	NA	NA	6	0.025
Lead	0.010 - 250	0.02	0.003	4	0.04	1	0.010
Magnesium	0.16 - 500	0.04	0.020	50	0.50	50	0.16

<sup>(1)</sup> Thermal Jarrell Ash (TJA) ICAP61E instrument: soil RL based on 1.00 gram of sample diluted to final volume of 100 mL (sample preparation method #3050); water RL based on sample preparation Methods #3015.

<sup>(2)</sup> Thermal Elemental Trace Analyzer instrument: soil RL based on 1.00 gram of sample diluted to final volume of 100 mL (sample preparation method #3050); water RL based on sample preparation Methods #3015.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
 PAGE: 20 of 42  
 REV: 3.0  
 DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

TABLE 1. ICP Detection and Reporting Limits for TAL Compounds (cont'd)							
				TYPICAL REPORTING LIMITS			
ANALYTE	TYPICAL ANALYTICAL RANGE (mg/L)	TYPICAL INSTRUMENT DETECTION LIMIT (IDL, mg/L)		ICAP61E <sup>(1)</sup>		Trace <sup>(2)</sup>	
		ICAP 61E	Trace	Soil (mg/kg)	Water (mg/L)	Soil (mg/kg)	Water (mg/L)
Manganese	0.002 - 25	0.0005	0.001	1	0.005	0.3	0.002
Molybdenum	0.010 - 50	NA	0.002	NA	NA	1	0.010
Nickel	0.005 - 100	0.005	0.001	1	0.01	0.5	0.005
Potassium	0.25 - 50	0.60	0.050	200	2	50	0.25
Selenium	0.010 - 100	0.08	0.003	14	0.14	1.1	0.010
Silver	0.004 - 100	0.003	0.001	0.5	0.005	0.5	0.004
Sodium	1.0 - 50	0.03	0.20	50	0.50	100	1.2
Thallium	0.014 - 100	0.04	0.003	10	0.08	2	0.014
Vanadium	0.003 - 100	0.003	0.001	2.0	0.01	0.4	0.003
Zinc	0.006 - 50	0.001	0.002	2.0	0.01	1	0.006
Iron 2714	0.03 - 300	NA	0.010	NA	NA	10	0.030

<sup>(1)</sup> Thermal Jarrell Ash (TJA) ICAP61E instrument: soil RL based on 1.00 gram of sample diluted to final volume of 100 mL (sample preparation method #3050); water RL based on sample preparation Methods #3015.

<sup>(2)</sup> Thermal Elemental Trace Analyzer instrument: soil RL based on 1.00 gram of sample diluted to final volume of 100 mL (sample preparation method #3050); water RL based on sample preparation Methods #3015.

NA = Not Applicable

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 21 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

TABLE 2. Typical ICP Calibration Standard Concentrations  
for TAL Compounds in Soil and Water Matrices

Volume of 100 mg/L Intermediate Cal Standard, mL	Volume of 1000 mg/L Intermediate Standard, mL	Volume of 1000 mg/L Stock Cal Standard, mL	Volume of 10000 mg/L Stock Cal Standard, mL	Elements	Final Working Standard Concentration, mg/L
10	-	-	-	Be, Cd, Cr Co, Cu, Mn, Ni, Ag, Zn	5
-	1	-	-	Ba, V	5
-	-	1	-	Sb	5
-	-	1	-	As	5
-	-	1	-	Se	5
-	-	1	-	Tl	5
-	-	1	-	Pb	5
-	-	1	-	Mo	5
-	-	-	1	Al	50
-	-	-	1	Ca	50
-	-	-	0.5	Fe	25
-	-	-	1	Mg	50
-	-	-	0.5	K	25
-	-	-	1	Na	50



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 22 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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APPENDIX B  
TJA ICAP-61E Daily Operation Procedures  
SOP #1811  
March 2006



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 23 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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The attached procedures summarize the REAC Inorganic Laboratory daily operation practice for sample analysis with the Thermo Jarrell Ash (TJA) Corporation model ICAP-61E ICP spectrometer. These procedures are based on the assumption that methods have already been developed and the instrument is in the standby mode. Refer to the TJA ICAP-61E operator's manual for specific details on instrument setup, methods development, operation, and maintenance.

### TJA ICAP-61E DAILY OPERATION PROCEDURES

**PURPOSE:** To provide a step by step procedure for the proper setup and operation of the TJA ICAP-61E instrument.

**REFERENCE:** Thermo Jarrell Ash Corporation, ICAP-61E Spectrometer Operator's Manual.

Assuming that methods have already been developed, the computer is Windows based, and the instrument is on standby mode, the normal daily operation of the ICAP-61E is as follows.

#### IGNITE THE TORCH

1. Set the argon pressure at the supply source to 80 - 90 psi. Set the argon purge level to five units on the spectrometer front panel. Press the instrument reset button on the spectrometer front panel.
2. Turn on the video display, printer, and computer (in that order). The computer automatically boots up into ThermoSPEC software, and the main menu is displayed.
3. Connect the sample pump winding on the peristaltic pump and place the sample line in 10% nitric acid solution.
4. Access the control panel by pressing <Ctrl> and <Panel> (function key, <F6>) simultaneously. The control panel is displayed.
5. Press <StartUp> (function key <F1>). The purge time should be set for 90 seconds. The startup power should be set to the value to be used in the first analysis. Set the plasma conditions to match the first analysis. Press <Continue> to ignite the plasma.
6. Allow the plasma to stabilize thermally for approximately 30 - 60 minutes before taking quantitative analytical data.

#### CHOOSE A METHOD

1. From the main menu, highlight **DEVELOPMENT** and enter the **Methods** module.
2. Press <F6> and enter the number of the method; press <ENTER> twice.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 24 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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3. OPTIONAL: Press <F1> to choose the elements to run or delete from the method.
4. OPTIONAL: Press <F9> to save.
5. OPTIONAL: Press <ElemInfo> (<F5>) and enter high, low offset; high std, low std; and concentration of elements in each standard.
6. OPTIONAL: Press <F9> to save.
7. Press <F3> to name the file for data storage (database file name) and to select the number of replicates (normally 4). Use the naming convention Sddmmyy for soil, Wddmmyy for water, and Tddmmyy for tissue samples where ddmmyy is the current date (day/month/year).
8. Press <F9> to save.
9. Press <F9> to save the entire method.
10. Press <ESC> to return to the main menu.
11. Select **OPERATIONS**, press <ENTER>.
12. Select **Analysis**, press <ENTER>.
13. The method selected is highlighted, press <ENTER>.

#### PROFILE

For optimum performance of the polychromator, each spectral line must be centered on its exit slit which is positioned before the photomultiplier tube. Maintaining this optical alignment during operation is called profiling. This is performed by introducing light to the system from a sample solution in the plasma. Only one spectral line is profiled; all other lines are preset relative to the profiled line.

1. Press <Profile> in the "Analysis" menu.
2. Aspirate a 10 mg/L copper standard solution for 30 seconds.
3. Press <F3> (<Automatic>), and then <F1> (<Run>).
4. After a minute or two, a table and graph appear on the screen; read the PEAK POSITION. The number should be plus or minus 0.20. If so, press <F9> to save the profile; if not, press <F1> to calculate a new micrometer position, escape from the bad profile (press <ESC>), press <ENTER>, and repeat step #3 above after adjusting the micrometer. Continue adjusting the micrometer until the PEAK POSITION is stable at plus or minus 0.20 units.



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 25 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### CALIBRATE THE INSTRUMENT:

1. Press <F3> to standardize.
2. Analyze the calibration blank, press <F9> to save.
3. After analyzing each standard, press <F9> to save.
4. After calibrating the instrument, press <F9> twice to store the calibration data.

#### TO ANALYZE SAMPLES (Manually):

1. Press <F1>, type in sample name and other information.
2. Wait five seconds after the sample reaches the nebulizer, press <F1> to start the analysis.
3. Repeat step 1 and 2 for every sample.
4. After every 10th sample, press <F2> (<QC Stdrd>) to analyze the Continuing Calibration Verification (CCV) standard.
5. After CCV analysis, press <F4> (<Blank>) to analyze the Continuing Calibration Blank (CCB) standard.

#### TO ANALYZE SAMPLES (With Autosampler):

1. From the main menu, select **OPERATION**, then *Autosampler set up*, press <ENTER>.
2. Type in the autosampler table name, press <ENTER>.
3. Press <F3> to select a table set.
4. Type in the appropriate information such as the method name, lab ID, rinse time, etc.
5. Press <F1> (<EdSample>) to edit or enter information about the samples in the set.
6. After every 10 sample analyses, press <F5> to insert the QC Check std. (CCV) and <F7> to insert the Blank Check std. (CCB).
7. Press <F8> to insert rinses.
8. Press <F9> twice to store the autosampler table.
9. Press <ESC>, select **OPERATION**, press <ENTER>; select *Analyze*, press <ENTER>; press <F9> for autosampler (be sure correct autosampler table has been recalled).

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 26 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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NOTE: Check rinse bottle, tubing, samples, and QC cups. Fill appropriate sample cups with standards and samples.

10. Press <F1> to activate autosampler.

#### TO PRINT RESULTS/GENERATE ASCII REPORT FILES:

1. From the main menu, select **Report Writer** in the "IMS" menu and press <ENTER>.
2. Follow the instructions in the "Selecting Report Contents" screen to specify filenames and the start/end Date Range. Press <F9> to continue with the "Edit Report Parameters" screen.
3. Specify the "Primary Sort" (for example, chronological) and the "Use Subset of Elements" fields as necessary. Press <F9> to print the report.
4. After the report is printed, the ThermoSPEC program returns to the "Selecting Report Contents" screen.
5. Press <F9> for the "Editing Report Parameters" screen. Select the **Send Report To:** field, press the space bar key, select **ASCII File**, and press <ENTER>. Press the down arrow key once for the **ASCII File Name:** field, type "z:mmddyy", press <ENTER>, and press <F9> to generate the file. **NOTE:** "mmddyy" is the month/day/year which was entered as the start date for the printed report. If a subset report is printed, "z:mmddyyS" is used for the corresponding ASCII file name ("S" denotes subset). The same file name **MUST NOT** be used for different ASCII file reports.
6. Return to the main menu after all reports and ASCII files have been generated.

#### TO ARCHIVE SAMPLE DATA:

1. From the main menu, select **Filer** in the "IMS" menu and press <ENTER>.
2. Select **12. Samples Archival** and press <ENTER> for the "Selecting Archive Choices" screen. Press <F1> to archive sample data and to continue with the "Selecting Archive Contents" screen. Press <F1> again to select the archive data storage path: type "I:mmddyy.arc" to store the archive data file (mmddyy.arc) on drive "I:" where, mmddyy is the current date (month/day/year).
3. Enter the start/end Date Range values as necessary (generally the current date). Specify the same values as those used to generate the printed report. Press <F9> to archive the data.
4. Return to the main menu after all data has been archived.
5. From the main menu, select **Quit to DOS** in the "Exit" menu and press <ENTER>.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 27 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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6. Backup all results and database files to the zip disk using Windows Explorer.
  - Put the zip disk in the drive on the ICAP61E computer (just below the CDROM drive).
  - Create a new folder on the zip disk with the current date, ie, the date that the samples were analyzed: ddmmyyyy (example 09042003 for 04 Sept 2003).
  - Copy the appropriate archive and RES1 files from drive "I:" to this zip disk folder.
  - Copy all files with the current date from drive "Z:" to this folder. NOTE, this will include ASCII and database files created/modified in this sample analysis run.
  - Verify that all files have been copied from drives "I:" and "Z:" to the appropriate zip disk folder.
  - Remove the zip disk from the drive.
  - Delete the "RES1" file from drive "I:".
7. After all files have been copied to the archive zip disk folder, close Windows Explorer. From the Windows Desktop START menu, select Shutdown, then Restart to restart the computer. This will restart the ThermoSPEC software.

#### TO SHUT DOWN INSTRUMENT (To standby mode):

1. Prior to shutdown, rinse with 10% nitric acid for at least 5 minutes followed by DI water for at least 5 minutes, then remove the sample tube and air dry for at least 2 minutes.
2. Press <Ctrl> and <F6> simultaneously to access the control panel screen.
3. Press <F7> (<Shutdown>) to turn off the plasma and the RF generator in an orderly fashion.
4. Loosen the sample pump winding on the peristaltic pump.
5. Reduce the argon pressure at the supply source to 10 - 20 psi.
6. Exit the ThermoSPEC software (this goes to the Windows Desktop). From the START menu, select Shutdown, then Shutdown again (in the Shutdown Windows box) to shut down the computer.

#### TROUBLESHOOTING

##### (SEE Section 19 of the Thermo Elemental ICAP-61E Analyzer Operator's Manual)

1. ICP will not start (see "IGNITE THE TORCH" above and Section 10 of the Manual)
  - Check for: power supplies are ON, Argon gas is ON and at correct pressure, cooling water circulator is ON, exhaust is ON, sample tubing is connected, correct tension on pump winding tubing, and no air leaks in Argon gas lines.
  - After making any necessary adjustments, try again.
  - If fails to start, power down, power up, reset, and try again.
  - If repeated attempts fail to start the ICP, call Thermo Electron Customer Support for

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 28 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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assistance.

2. High RSD During Standardization (STD2 RSD > 2%)
  - Repeat the measurement.
  - Adjust tension on the Sample pump winding tubing and repeat.
  - Check and replace if necessary: sample line capillary tubing, sample uptake wand connector tubing, and/or sample uptake wand.
  - Re-standardize as needed or based on professional judgement.
3. Initial QC Samples Fail
  - Repeat the measurement.
  - Check for high RSD for results.
  - Check for contamination.
  - Check another QC sample.
  - Check for profile drift; if profile has drifted (peak position outside 0 +/- 0.2), reset profile and re-standardize.
4. Sample Analysis Problems
  - A. CCV and/or CCB results pass (but indicate drift based on professional judgement).
    - check for blockage in the sample wand or tubing; replace as needed and re-standardize.
    - check for profile drift every 8 hours or as needed (peak position outside 0 +/- 0.2), reset profile and reanalyze CCV and CCB. If they pass continue with analysis, otherwise re-standardize.
  - C. CCV Fails
    - check for high RSD (>3%) and repeat the measurement.
    - If fails again, check for contamination, profile drift; correct problems as necessary and re-standardize.
  - D. CCB Fails
    - Check for carryover; if so, wait a minimum of 5 minutes.
    - Repeat the measurement.
    - If fails again, check for contamination, profile drift; correct problems as necessary and re-standardize.
  - E. Re-standardize as necessary.



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 29 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### APPENDIX C

Thermo Elemental ICAP-61E Trace Analyzer Daily Operation Procedures

SOP #1811

March 2006



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 30 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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The attached procedures summarizes the REAC Inorganic Laboratory daily operation practice for sample analysis with the Thermo Elemental model ICAP-61E Trace Analyzer ICP spectrometer (Trace). These procedures are based on the assumption that methods have already been developed and the instrument is in the standby mode. Refer to the Thermo Elemental ICAP-61E Trace Analyzer operator's manual for specific details on instrument setup, methods development, operation, and maintenance.

### THERMO ELEMENTAL ICAP-61E TRACE ANALYZER DAILY OPERATION PROCEDURES

**PURPOSE:** To provide a step by step procedure for the proper setup and operation of the Thermo Elemental ICAP-61E Trace Analyzer instrument.

**REFERENCE:** Thermo Elemental ICAP-61E Trace Analyzer Operator's Manual.

Assuming that methods have already been developed, the computer is Windows based, and the instrument is on standby mode, the normal daily operation of the Trace is as follows.

#### IGNITE THE TORCH

1. Set the argon pressure at the supply source to 80 - 90 psi. Set the argon purge level to 2 (two) units on the spectrometer front panel. Press the instrument reset button on the spectrometer front panel.
2. Turn on the video display, printer, and computer (in that order). The computer boots up into the Windows NT desktop. Click the "Run Trace" icon, and the main menu is displayed.
3. Connect the sample and internal standard (IS) pump windings on the peristaltic pump. Place the sample line in 10% nitric acid solution and the IS line in the internal standard solution.
4. Access the control panel by pressing <Ctrl> and <Panel> (function key, <F6>) simultaneously. The control panel is displayed.
5. Press <StartUp> (function key <F1>). The purge time should be set for 90 seconds. The startup power should be set to the value to be used in the first analysis. Set the plasma conditions to match the first analysis. Press <Continue> to ignite the plasma.
6. Allow the plasma to stabilize thermally for approximately 30 - 60 minutes before taking quantitative analytical data.

#### CHOOSE A METHOD

1. From the main menu, highlight **DEVELOPMENT** and enter the **Methods** module.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 31 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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2. Press <F6> and enter the number of the method; press <ENTER> twice.
3. OPTIONAL: Press <F1> to choose the elements to run or delete from the method.
4. OPTIONAL: Press <F9> to save.
5. OPTIONAL: Press <ElemInfo> (<F5>) and enter high, low offset; high std, low std; and concentration of elements in each standard.
6. OPTIONAL: Press <F9> to save.
7. Press <F3> to name the file for data storage (database file name). Use the naming convention; Tddmmyy where dddmmyy is the current date (day/month/year). Always begin the database name with the letter "T" (for "trace"), DO NOT begin with a number.
8. Press <F9> to save.
9. Press <F9> to save the entire method.
10. Press <ESC> to return to the main menu.
11. Select **OPERATIONS**, press <ENTER>.
12. Select **Analysis**, press <ENTER>.
13. The method selected is highlighted, press <ENTER>.

#### PROFILE

For optimum performance of the polychromator, each spectral line must be centered on its exit slit which is positioned before the photomultiplier tube. Maintaining this optical alignment during operation is called profiling. This is performed by introducing light to the system from a sample solution in the plasma. Only one spectral line is profiled; all other lines are preset relative to the profiled line.

1. Press <Profile> in the "Analysis" menu.
2. Aspirate a 5 mg/L arsenic standard (for profiling) solution for 30 seconds.
3. Press <F3> (<Automatic>), and then <F1> (<Run>).
4. After a minute or two, a table and graph appear on the screen; read the PEAK POSITION. The number should be plus or minus 0.20. If so, press <F9> to save the profile; if not, press <F1> to calculate a new micrometer position, escape from the bad profile (press <ESC>), press <ENTER>, and repeat step #3 above after adjusting the micrometer. Continue

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 32 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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adjusting the micrometer until the PEAK POSITION is stable at plus or minus 0.20 units.

#### CALIBRATE THE INSTRUMENT:

1. Press <F3> to standardize.
2. Analyze the calibration blank, press <F9> to save.
3. After analyzing each standard, press <F9> to save.
4. After calibrating the instrument, press <F9> twice to store the calibration data.

#### TO ANALYZE SAMPLES (Manually):

1. Press <F1>, type in sample name and other information.
2. Wait five seconds after the sample reaches the nebulizer, press <F1> to start the analysis.
3. Repeat step 1 and 2 for every sample.
4. After every 10th sample, press <F2> (<QC Stndrd>) to analyze the Continuing Calibration Verification (CCV) standard.
5. After CCV analysis, press <F4> (<Blank>) to analyze the Continuing Calibration Blank (CCB) standard.

#### TO ANALYZE SAMPLES (Autosampler):

1. From the main menu, select **OPERATION**, then *Autosampler set up*, press <ENTER>.
2. Type in the autosampler table name, press <ENTER>.
3. Press <F3> to select a table set.
4. Type in the appropriate information such as the method name, lab ID, rinse time, etc.
5. Press <F1> (<EdSample>) to edit or enter information about the samples in the set.
6. After every 10 sample analyses, press <F5> to insert the QC Check std. (CCV) and <F7> to insert the Blank Check std. (CCB).
7. Press <F8> to insert rinses.
8. Press <F9> twice to store the autosampler table.



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 33 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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9. Press <ESC>, select **OPERATION**, press <ENTER>; select **Analyze**, press <ENTER>; press <F9> for autosampler (be sure correct autosampler table has been recalled).  
NOTE: Check rinse bottle, tubing, samples, and QC cups. Fill appropriate sample cups with standards and samples.
10. Press <F1> to activate autosampler.

#### TO ANALYZE SAMPLES (Software Supervised Autosampler):

1. Make sure that the serial cable **IS NOT** connected to the autosampler. From the main menu, select **OPERATION**, then **Autosampler set up**, press <ENTER>.
2. Select the appropriate autosampler table, press <ENTER>. NOTE: tables are setup for initial QC runs for both soil and water as well as the final QC run.
3. Press <F1> to activate autosampler.
4. The software will attempt to run the autosampler and will give a message that it is not operating properly. Press <ENTER> to continue when you see the message; you will be prompted for the proper QC sample to run.

#### TO PRINT RESULTS/GENERATE ASCII REPORT FILES:

1. From the main menu, select **Report Writer** in the "IMS" menu and press <ENTER>.
2. Follow the instructions in the "Selecting Report Contents" screen to specify filenames and the start/end Date Range. Press <F9> to continue with the "Edit Report Parameters" screen.
3. Specify the "Primary Sort" (for example, chronological) and the "Use Subset of Elements" fields as necessary. Press <F9> to print the report.
4. After the report is printed, the ThermoSPEC program returns to the "Selecting Report Contents" screen.
5. Press <F9> for the "Editing Report Parameters" screen. Select the **Send Report To:** field, press the space bar key, select **ASCII File**, and press <ENTER>. Press the down arrow key once for the **ASCII File Name:** field, type "z:mmddyy", press <ENTER>, and press <F9> to generate the file. NOTE: "mmddyy" is the month/day/year which was entered as the start date for the printed report. If a subset report is printed, "z:mmddyyS" is used for the corresponding ASCII file name ("S" denotes subset). The same file name **MUST NOT** be used for different ASCII file reports.
6. Return to the main menu after all reports and ASCII files have been generated.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 34 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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#### TO ARCHIVE SAMPLE DATA:

1. From the main menu, select **Filter** in the "IMS" menu and press <ENTER>.
2. Select **12. Samples Archival** and press <ENTER> for the "Selecting Archive Choices" screen. Press <F1> to archive sample data and to continue with the "Selecting Archive Contents" screen. Press <F1> again to select the archive data storage path: type "D:mmddyy.arc" to store the archive data file (mmddyy.arc) on drive "D:" where, mmddyy is the current date (month/day/year).
3. Enter the start/end Date Range values as necessary (generally the current date). Specify the same values as those used to generate the printed report. Press <F9> to archive the data.
4. Return to the main menu after all data has been archived.
5. From the main menu, select **Quit to DOS** in the "Exit" menu and press <ENTER>.
6. Backup all results and database files to the Rewritable (RW) CD using Windows Explorer.
  - Put the CD in the CD drive on the Trace NT computer; wait until you see the message that the drive is ready, then click OK.
  - Create a new folder on the CD with the current date, ie, the date that the samples were analyzed: ddmmYYYY (example 09032004 for 03 Sept 2004). Create this folder in the appropriate subfolder on the CD.
  - Copy the appropriate archive and RES1 files from drive "D:" to this CD folder.
  - Copy all files with the current date from drive "Z:" to this folder. NOTE, this will include ASCII and database files created/modified in this sample analysis run.
  - Verify that all files have been copied from drives "D:" and "Z:" to the appropriate CD folder.
  - Eject the CD from the drive.
  - Delete the "RES1" file from drive "D:".
7. After all files have been copied to the archive CD folder, close Windows Explorer. From the Windows Desktop, click the "Run Trace" icon to restart the ThermoSPEC software.

#### TO SHUT DOWN INSTRUMENT (To standby mode):

1. Prior to shutdown, place both sample and IS lines in 10% nitric acid and rinse for at least 5 minutes followed by DI water for at least 5 minutes, then remove both lines and air dry for at least 2 minutes.
2. Press <Ctrl> and <F6> simultaneously to access the control panel screen.
3. Press <F7> (<Shutdown>) to turn off the plasma and the RF generator in an orderly fashion.
4. Loosen the sample and IS pump windings on the peristaltic pump.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 35 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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5. Reduce the argon pressure at the supply source to 10 - 20 psi.
6. Exit the ThermoSPEC software (this goes to the Windows Desktop). From the START menu, select Shutdown, then Shutdown again (in the Shutdown Windows box) to shut down the computer.

### TROUBLESHOOTING

(SEE Section 19 of the Thermo Elemental ICAP-61E Trace Analyzer Operator's Manual)

1. ICP will not start (see "IGNITE THE TORCH" above and Section 10 of the Manual)
  - Check for: power supplies are ON, Argon gas is ON and at correct pressure, cooling water circulator is ON, exhaust is ON, sample and IS tubing is connected, correct tension on pump winding tubing, and no air leaks in Argon gas lines.
  - After making any necessary adjustments, try again.
  - If fails to start, power down, power up, reset, and try again.
  - If repeated attempts fail to start the ICP, call Thermo Electron Customer Support for assistance.
2. High RSD During Standardization (IS RSD > 3% and/or STD2 RSD > 2%)
  - Repeat the measurement.
  - Adjust tension on the IS and Sample pump winding tubing and repeat.
  - Check and replace if necessary: IS mix kit tubing, sample line capillary tubing, sample uptake wand connector tubing, IS wand connector tubing, sample wand, and/or IS uptake wand.
  - Re-standardize as needed or based on professional judgement.
3. Initial QC Samples Fail
  - Repeat the measurement.
  - Check for high IS RSD and high RSD for results.
  - Check for contamination.
  - Check another QC sample.
  - Check for profile drift; if profile has drifted (peak position outside 0 +/- 0.2), reset profile and re-standardize.
4. Sample Analysis Problems
  - A. High RSD for IS (>3%), see above.
  - B. IS counts, CCV, and/or CCB results pass (but indicate drift based on professional judgement).
    - check for blockage in the IS and/or sample wand or tubing; replace as needed and re-standardize.

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 36 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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- check for profile drift every 8 hours or as needed (peak position outside 0 +/- 0.2), reset profile and reanalyze CCV and CCB. If they pass continue with analysis, otherwise re-standardize.

C. CCV Fails

- repeat the measurement.  
- If fails again, check for high IS RSD, IS drift, profile drift; correct problems as necessary and re-standardize.

D. CCB Fails

- Check for carryover; if so, wait a minimum of 5 minutes.  
- Repeat the measurement.  
- If fails again, check for high IS RSD, IS drift, profile drift; correct problems as necessary and re-standardize.

E. Re-standardize as necessary based on professional judgement.



## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 37 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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APPENDIX D  
Attachments  
SOP #1811  
March 2006



Response Engineering and Analytical Contract

## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 38 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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ATTACHMENT 1  
Water and Soil RL Spiking Solution  
SOP #1811  
March 2006

## Standard Preparation Log

Name: Water Reporting Level (RL) Spike solution (0.5 mL to 500 mL final volume)

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	REAC Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		40	0.4
2	1000	Sb		14	1.4
3	1000	As		15	1.5
4	1000	Ba		2	0.2
5	1000	Be		2	0.2
6	1000	Cd		2	0.2
7	10000	Ca		63	0.63
8	1000	Cr		2	0.2
9	1000	Co		3	0.3
10	1000	Cu		4	0.4
11	10000	Fe		25	0.25
12	1000	Pb		10	1
13	10000	Mg		160	1.6
14	1000	Mn		2	0.2
15	1000	Mo		10	1
16	1000	Ni		5	0.5
17	10000	K		250	2.5
18	1000	Se		10	1
19	1000	Ag		4	0.4
20	10000	Na		1200	12
21	1000	Tl		14	1.4
22	1000	V		3	0.3
23	1000	Zn		6	0.6

TOTAL 28.18

## Standard Preparation Log

Name: Soil Reporting Level (RL) Spike solution (0.5 mL to 500 mL final volume)

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	REAC Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		250	2.5
2	1000	Sb		14	1.4
3	1000	As		20	2
4	1000	Ba		3	0.3
5	1000	Be		3	0.3
6	1000	Cd		4	0.4
7	10000	Ca		200	2
8	1000	Cr		4	0.4
9	1000	Co		3	0.3
10	1000	Cu		4	0.4
11	10000	Fe		60	0.6
12	1000	Pb		10	1
13	10000	Mg		500	5
14	1000	Mn		3	0.3
15	1000	Mo		10	1
16	1000	Ni		5	0.5
17	10000	K		500	5
18	1000	Se		11	1.1
19	1000	Ag		5	0.5
20	10000	Na		1000	10
21	1000	Tl		20	2
22	1000	V		4	0.4
23	1000	Zn		10	1

TOTAL 38.4





## STANDARD OPERATING PROCEDURES

SOP: 1811  
PAGE: 41 of 42  
REV: 3.0  
DATE: 03/17/06

### DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) METHODS (EPA/SW-846 Methods 3015/3050B/6010B)

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ATTACHMENT 2  
Spiking Solutions  
SOP #1811  
March 2006

# REAC METAL LABORATORY

## SPIKING SOLUTION LOG

SOLUTION	CONCENTRATION mg/l	PARAMETERS	DATE PREPARED	LOT#	Expiration Date	PREPARED BY
SPIKING SOLN. 1 (61E TRACE-SOIL)	20 ppm	Ag, Be, Cd, Cr, Cu, Co, Mn, Ni,	10/18/05	MI10180501	03/06	C. Gasser
	20 ppm	Zn, Ba, V, As, Sb, Pb, Mo				
	10 ppm	Se, Tl				
	100 ppm	Al, Ca, Fe, Mg, K				
	400 ppm	Na				
SPIKING SOLN. 2 (61E TRACE-WATER)	10 ppm	Ag, Be, Cd, Cr, Cu, Co, Mn, Ni,	10/18/05	MI10180502	03/06	C. Gasser
	10 ppm	Zn, Ba, V, Sb, Mo				
	5 ppm	As, Se, Tl, Pb				
	100 ppm	Al, Ca, Fe, Mg, K				
	400 ppm	Na				
SPIKING SOLN. 3 (Wipe/Air Spiking Soln.)						
	100 ppm	Al, Ca, Fe, Mg, K, Na	09/21/05	MI09210501	02/06	D. Kalnicky
	5 ppm	Ag, Be, Cd, Cr, Cu, Co, Mn, Ni,				
	5 ppm	Zn, Ba, V, Mo, As, Se, Tl, Sb, Pb				

Revised: 10/20/05

## STANDARD OPERATING PROCEDURE (SOP) CHANGE FORM

This change form will be used to document any required and/or needed changes in the SOP prior to the next revision. If the SOP will not be revised immediately, this form will be attached to the current version of the SOP to alert users/viewers of any changes. Changes will not take effect until all required signatures are present. This form must be forwarded to the Quality Assurance Officer for incorporation into the next revision of the SOP and as an attachment to the current approved SOP.

SOP Number: 1811

SOP Title: Determination of Metals by Inductively Coupled Plasma (ICP) Methods

SOP Revision Number: 3.0

### Requested Changes:

1. Add to last paragraph of section IV, page 4, "Interferences and Potential Problems":

ICP analyzers may have a hydroxyl band interference for aluminum measurement. Due to this interference, the Reporting Level (RL) for aluminum is typically elevated to 0.050 mg/L or higher depending on the sample matrix type.

2. Appendix A, Table 1

Change the RLs for the analytes listed in the attached table (see attached table).

3. Appendix D, Attachment 1

Replace Standard Preparation Logs for water and soil reporting level spike solutions with the attached pages (see 2 attached prep logs).

Requestor: \_\_\_\_\_

REAC Group Leader: \_\_\_\_\_

REAC Section Leader: \_\_\_\_\_

REAC Quality Assurance Officer: \_\_\_\_\_

Date Approved (by QAO): \_\_\_\_\_

Changes to ICP Reporting Limits for TAL Compounds				
ANALYTE	TYPICAL REPORTING LIMITS			
	ICP Trace <sup>(2)</sup>			
	Soil (mg/kg)		Water (mg/L)	
	From	To	From	To
Aluminum	25	20	0.040	0.050
Arsenic	2	1.5	0.015	0.016
Barium	0.3	0.4	0.002	NA
Cadmium	0.4	NA	0.002	0.003
Calcium	20	9.9	0.063	0.060
Chromium	0.4	0.5	0.002	0.003
Cobalt	0.3	0.4	0.003	NA
Iron 2599	6	9.9	0.025	NA
Magnesium	50	20	0.16	NA
Manganese	0.3	0.4	0.002	NA
Molybdenum	1	0.7	0.010	0.006
Nickel	0.5	0.6	0.005	NA
Potassium	50	25	0.25	0.20
Selenium	1.1	1.3	0.010	NA
Thallium	2	1.7	0.014	0.016
Zinc	1	2.3	0.006	NA
Iron 2714	10	15	0.030	0.060

<sup>(2)</sup> Thermal Elemental Trace Analyzer instrument: soil RL based on 1.00 gram of sample diluted to final volume of 100 mL (sample preparation method #3050); water RL based on sample preparation Methods #3015.

NA - Not Applicable

## Standard Preparation Log

Name: Water Reporting Level (RL) Spike solution \*

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	REAC Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		50	0.5
2	1000	Sb		14	1.4
3	1000	As		16	1.6
4	1000	Ba		2	0.2
5	1000	Be		2	0.2
6	1000	Cd		3	0.3
7	10000	Ca		60	0.6
8	1000	Cr		3	0.3
9	1000	Co		3	0.3
10	1000	Cu		4	0.4
11	10000	Fe		25	0.25
12	1000	Pb		10	1
13	10000	Mg		160	1.6
14	1000	Mn		2	0.2
15	1000	Mo		6	0.6
16	1000	Ni		5	0.5
17	10000	K		200	2
18	1000	Se		10	1
19	1000	Ag		4	0.4
20	10000	Na		1200	12
21	1000	Tl		16	1.6
22	1000	V		3	0.3
23	1000	Zn		6	0.6

\* RLW - Water RL solution (0.5 mL spike solution to 500 mL final volume)

**TOTAL 27.85**

45

## Standard Preparation Log

Name: Soil Reporting Level (RL) Spike solution \*

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	REAC Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		200	2
2	1000	Sb		14	1.4
3	1000	As		15	1.5
4	1000	Ba		4	0.4
5	1000	Be		3	0.3
6	1000	Cd		4	0.4
7	10000	Ca		99	0.99
8	1000	Cr		5	0.5
9	1000	Co		4	0.4
10	1000	Cu		4	0.4
11	10000	Fe		99	0.99
12	1000	Pb		10	1
13	10000	Mg		200	2
14	1000	Mn		4	0.4
15	1000	Mo		7	0.7
16	1000	Ni		6	0.6
17	10000	K		250	2.5
18	1000	Se		13	1.3
19	1000	Ag		5	0.5
20	10000	Na		1000	10
21	1000	Tl		17	1.7
22	1000	V		4	0.4
23	1000	Zn		23	2.3

\* RLS - Soil RL solution (0.5 mL spike solution to 500 mL final volume)

TOTAL 32.68